#### FOODBALT 2008

# ALUMINIUM ANALYSIS – A PROFICIENCY TESTING STUDY

# Amanda Earnshaw, Laura Prenton, Linda Owen

FAPAS<sup>®</sup>, Central Science Laboratory, Sand Hutton, York, YO41 1LZ, UK

#### Abstract

Proficiency testing is an external analytical quality assurance (AQA) measure i.e. the quality of the analytical result is checked against criteria that are set independently of the laboratory carrying out the analyses. In a proficiency test (PT), the participants' results are used to derive the assigned value. Then, the difference between each result and the assigned value is compared to the target standard deviation. The end product of the performance assessment is a standardised statistic known as a z-score.

In addition to assessing performance of participating laboratories, proficiency testing highlights problems in laboratory analysis and can be used as an educational tool to help to improve data quality. The last three FAPAS<sup>®</sup> proficiency tests (0770, 0787, 0784) have highlighted problems with assessing aluminium.

The results from laboratories taking part in a typical chemical analysis will be normally distributed i.e. the majority of results will be centred on a mean value. In proficiency test 0770 (soya flour), multiple modes were observed rather than a normal distribution. As there was insufficient evidence to draw any conclusions it was not possible to set an assigned value or calculate any z-scores for this analyte. FAPAS<sup>®</sup> test 0784 (milk powder) had a bimodal distribution and 3 modes were observed in the FAPAS<sup>®</sup> test 0787 (soya flour). In these tests, the major mode was used as the assigned value.

FAPAS<sup>®</sup> investigated using a reference value to check the assigned value for one of the proficiency tests (0787). This reference value was compared to the results of the proficiency test and the methods used by participants. The reference value was close to the major mode and the results for ICP-MS methods were also similar to the reference value.

Key words: proficiency testing, aluminium, quality

### Introduction

Plants can take up aluminium from the soil and from water. So some plants, such as tea, and some herbs and leafy vegetables, can build up high levels of aluminium naturally. Aluminium can also be added to food during processing and some food additives contain aluminium. These are used in foods such as bakery products, dried powdered foods and drinks, and processed cheeses to improve the texture (Pennington, 1988).

It is therefore important that materials, such as aluminium, that are added to food or come into contact with food, do not make food harmful and that it does not change the nature, substance or quality of the food.

Scientific judgements on food data quality are under continuous scrutiny. It is therefore important to demonstrate that adequate confidence can be placed on results obtained by laboratories. Laboratories need, therefore, to demonstrate their performance and reliability in such analyses. It is widely accepted in most areas of food analysis and regulation that there are a number of essential elements to laboratory quality assurance. These elements include the use of validated analytical methods, accreditation involving third party auditing and the participation in laboratory proficiency testing schemes (Wood, Nilsson and Wallin, 1998). Thus, laboratories take part in proficiency testing for different reasons, which include export regulation on contaminants, labelling regulations, customer requirements for quality and quality data for food databases.

There is an increasing demand for independent proof of competence both from regulatory bodies and customers. Individual laboratories need to know how well they perform against objective standards and how their analytical results compare with others.

By setting the acceptable allowable variation around the assigned value at a level that reflects best practice PT testing provides such objective standards. By expressing the participants' submitted results as z-scores they can be compared with each other, with those at the extremes of the overall distribution being clearly indicated.

### FOODBALT 2008

value, the uncertainty of the mode was larger than expected and had a questionable effect on participants' z-scores. Hence the assigned value and z-scores were issued for information only. The homogeneity mean value was 973  $\mu$ g kg<sup>-1</sup> and was similar to the major mode.



Figure 2: Bump-hunting Histogram of Aluminium in FAPAS<sup>®</sup> 0784 Proficiency Test

The recent soya flour PT (0787; FAPAS<sup>®</sup> 2007b) identified three modes (Figure 3). The major mode (6.81  $\mu$ g kg<sup>-1</sup>) was used to set the assigned value, but z-scores were again issued for information only. The homogeneity mean value for aluminium was 7.1  $\mu$ g kg<sup>-1</sup>.



Figure 3: Bump-hunting Histogram of Aluminium in FAPAS® 0787 Proficiency Test

Figure 4 is a dot plot, from PT 0787, of the valid submitted results for aluminium, separated by participants' declared methodology. The seven ICP-MS results (5.98-9.496  $\mu$ g kg<sup>-1</sup>) were clustered around the assigned value (6.81  $\mu$ g kg<sup>-1</sup>), whilst data from other detection methods appear to be more widely scattered (between 3 and 35  $\mu$ g kg<sup>-1</sup>)

FOODBALT 2008



Figure 4: Methods used for Aluminium Analysis in FAPAS<sup>®</sup> 0787 Proficiency Test

A UK national reference laboratory for metallic contaminants undertook some research to determine the reference value for aluminium in soya flour (0787). The content of Al was determined using ICP-MS and was found to be 7.22  $\mu$ g kg<sup>-1</sup>. This value was close to the homogeneity mean value and the major mode, providing evidence that the major mode can be used as the assigned value for aluminium proficiency tests.

# Conclusions

Aluminium analysis is of particular importance in monitoring foods to ensure that there are not harmful levels present or that the quality of food is not compromised. Thus, ensuring that data quality is luminium is prone to contamination from the laboratory environment e.g. from traces in acids, leaching from glassware and from powdered gloves. However aluminium can be difficult to solubilise when digesting a solid sample, so under reporting can occur which may be defit for purpose is important for end users. Proficiency testing is able to highlight problems in the quality of analytical results and consequently help to improve it. Analysis of apendent on matrix, method or aluminium species in the sample.

# Acknowledgements

The UK Food Standards Agency funded research into obtaining the reference value for aluminium in soya flour.

# References

- 1. Analytical Methods Committee, (1989) Robust statistics How not to reject outliers Part 1. Basic concepts, *Analyst*, *114*, pp. 1693–1697.
- 2. FAPAS<sup>®</sup> (2002) Protocol for the Food Analysis Performance Assessment Scheme, Organisation and Analysis of Data, 6th Edition. Central Science Laboratory, York, UK.
- 3. FAPAS<sup>®</sup> (2006) FAPAS<sup>®</sup> Proficiency Test 0770: Metallic Contaminants in Soya Flour: August October 2006. Central Science Laboratory, York, UK.
- 4. FAPAS<sup>®</sup> (2007a) FAPAS<sup>®</sup> Proficiency Test 0784: Metallic Contaminants in Milk Powder: May June 2007. Central Science Laboratory, York, UK.
- 5. FAPAS<sup>®</sup> (2007b) FAPAS<sup>®</sup> Proficiency Test 0789: Metallic Contaminants in Soya Flour: July August 2007. Central Science Laboratory, York, UK.
- 6. Fearn, T. & Thompson, M., (2001). A new test for sufficient homogeneity, Analyst, 126, pp. 1414–1417.
- Lowthian, P.J. & Thompson, M., 2002, Bump-hunting for the proficiency tester searching for multimodality, *Analyst*, 127, pp. 1359–1364
- 8. Pennington, J.A.T (1988) Aluminium content of foods and diets. Food Addit. Contam., 5, pp. 161–232.
- 9. Thompson, M., Ellison, S.L.R., & Wood, R., (2006) The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, *Pure Appl. Chem.*, 78(1), pp. 145–196.
- 10. Wood, R., Nilsson, A. & Wallin, H. (1998) *Quality in the Food Analysis Laboratory*. The Royal Society of Chemistry, Cambridge, UK.